

# Filled $\text{Co}_x\text{Ni}_{4-x}\text{Sb}_{12-y}\text{Sn}_y$ skutterudites: processing and thermoelectric properties

**Jon Mackey**

Mechanical Engineering,  
University of Akron

**Alp Sehirlioglu**

Materials Science and Engineering,  
Case Western Reserve University

**Fred Dynys**

NASA Glenn Research Center

NASA Cooperative Agreement: NNX08AB43A

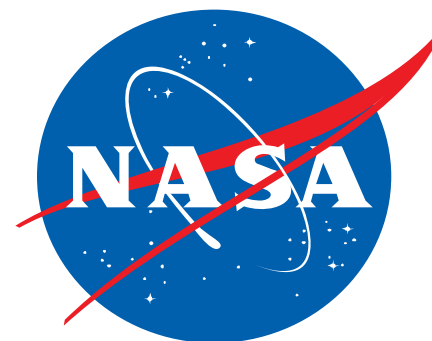
NASA/USRA Contract: 04555-004

The  
University  
of Akron



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UNIVERSITY EST. 1826

think beyond the possible



# Introduction

# Processing

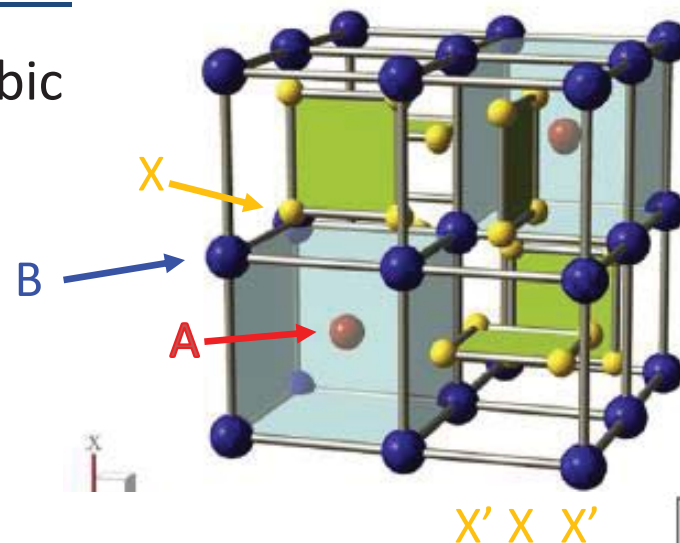
# Properties

## System Background

- Skutterudites are based on  $\text{CoAs}_3$  mineral; first mined in Skutterud, Norway.
- Exhibit a high figure of merit for n-type systems ( $ZT=1.7$ ).
- Relatively low cost system.
- Introduce disorder on pnictogen ring sites (X).
  - Dominate heat carrying modes are associated with pnictogen vibration.
- Introduce a range of fillers (A) to scatter various phonon wavelengths.
- Tune electronic properties (A,B,X) for optimal thermoelectric power factor .

# Crystal Structure

## Body-centered cubic space group $Im\bar{3}$



Hydrogen 1 H 1.00794

Helium 2 He 4.00260

Lithium 3 Li 6.941

Beryllium 4 Be 9.0122

Boron 5 B 10.811

Carbon 6 C 12.011

Nitrogen 7 N 14.0064

Oxygen 8 O 15.9994

Fluorine 9 F 18.9984

Neon 10 Ne 20.1798

Sodium 11 Na 22.98976928

Magnesium 12 Mg 24.304

Aluminum 13 Al 26.9815386

Silicon 14 Si 28.0855

Phosphorus 15 P 30.973762

Sulfur 16 S 32.06

Chlorine 17 Cl 35.453

Argon 18 Ar 39.948

Potassium 19 K 39.0983

Calcium 20 Ca 40.078

Scandium 21 Sc 44.955912

Titanium 22 Ti 47.867

Vanadium 23 V 50.9415

Chromium 24 Cr 51.9961

Manganese 25 Mn 54.938045

Iron 26 Fe 55.845

Cobalt 27 Co 58.933195

Nickel 28 Ni 58.6934

Copper 29 Cu 63.546

Zinc 30 Zn 65.38

Gallium 31 Ga 69.723

Germanium 32 Ge 72.64

Arsenic 33 As 74.9216

Selenium 34 Se 78.96

Bromine 35 Br 79.904

Krypton 36 Kr 83.80

Rubidium 37 Rb 85.4678

Sr 87.62

Yttrium 39 Y 88.90584

Zirconium 40 Zr 91.224

Niobium 41 Nb 92.906

Molybdenum 42 Mo 95.94

Technetium 43 Tc [98]

Ruthenium 44 Ru 101.07

Rhodium 45 Rh 102.91

Palladium 46 Pd 106.42

Silver 47 Ag 107.8682

Cadmium 48 Cd 112.411

Indium 49 In 114.818

Tin 50 Sn 118.710

Antimony 51 Sb 121.757

Tellurium 52 Te 127.6

Iodine 53 I 126.905

Xenon 54 Xe 131.29

Cesium 55 Cs 132.905

Barium 56 Ba 137.327

Lanthanum 57 La 138.90547

Cerium 58 Ce 140.12

Praseodymium 59 Pr 140.90768

Neodymium 60 Nd 144.242

Europium 61 Eu 151.964

Gadolinium 62 Gd 157.25

Terbium 63 Tb 158.92535

Dysprosium 64 Dy 162.50015

Ytterbium 65 Yb 173.045

Lutetium 66 Lu 174.967

Hafnium 67 Hf 178.49

Tantalum 68 Ta 180.94788

Vanadium 69 V 50.9415

Chromium 70 Cr 51.9961

Manganese 71 Mn 54.938045

Iron 72 Fe 55.845

Cobalt 73 Co 58.933195

Nickel 74 Ni 58.6934

Copper 75 Cu 63.546

Zinc 76 Zn 65.38

Gallium 77 Ga 69.723

Germanium 78 Ge 72.64

Arsenic 79 As 74.9216

Selenium 80 Se 78.96

Bromine 81 Br 79.904

Krypton 82 Kr 83.80

Rubidium 83 Rb 85.4678

Sr 87.62

Yttrium 84 Y 88.90584

Zirconium 85 Zr 91.224

Niobium 86 Nb 92.906

Molybdenum 87 Mo 95.94

Technetium 88 Tc [98]

Ruthenium 89 Ru 101.07

Rhodium 90 Rh 102.91

Palladium 91 Pd 106.42

Silver 92 Ag 107.8682

Cadmium 93 Cd 112.411

Indium 94 In 114.818

Tin 95 Sn 118.710

Antimony 96 Sb 121.757

Tellurium 97 Te 127.6

Iodine 98 I 126.905

Xenon 99 Xe 131.29

Cesium 100 Cs 132.905

Barium 101 Ba 137.327

Lanthanum 102 La 138.90547

Cerium 103 Ce 140.12

Praseodymium 104 Pr 140.90768

Neodymium 105 Nd 144.242

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Manganese 116 Mn 54.938045

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Molybdenum 132 Mo 95.94

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Ruthenium 134 Ru 101.07

Rhodium 135 Rh 102.91

Palladium 136 Pd 106.42

Silver 137 Ag 107.8682

Cadmium 138 Cd 112.411

Indium 139 In 114.818

Tin 140 Sn 118.710

Antimony 141 Sb 121.757

Tellurium 142 Te 127.6

Iodine 143 I 126.905

Xenon 144 Xe 131.29

Cesium 145 Cs 132.905

Barium 146 Ba 137.327

Lanthanum 147 La 138.90547

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Europium 151 Eu 151.964

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Hafnium 157 Hf 178.49

Tantalum 158 Ta 180.94788

Vanadium 159 V 50.9415

Chromium 160 Cr 51.9961

Manganese 161 Mn 54.938045

Iron 162 Fe 55.845

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Germanium 168 Ge 72.64

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Krypton 172 Kr 83.80

Rubidium 173 Rb 85.4678

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Yttrium 174 Y 88.90584

Zirconium 175 Zr 91.224

Niobium 176 Nb 92.906

Molybdenum 177 Mo 95.94

Technetium 178 Tc [98]

Ruthenium 179 Ru 101.07

Rhodium 180 Rh 102.91

Palladium 181 Pd 106.42

Silver 182 Ag 107.8682

Cadmium 183 Cd 112.411

Indium 184 In 114.818

Tin 185 Sn 118.710

Antimony 186 Sb 121.757

Tellurium 187 Te 127.6

Iodine 188 I 126.905

Xenon 189 Xe 131.29

Cesium 190 Cs 132.905</

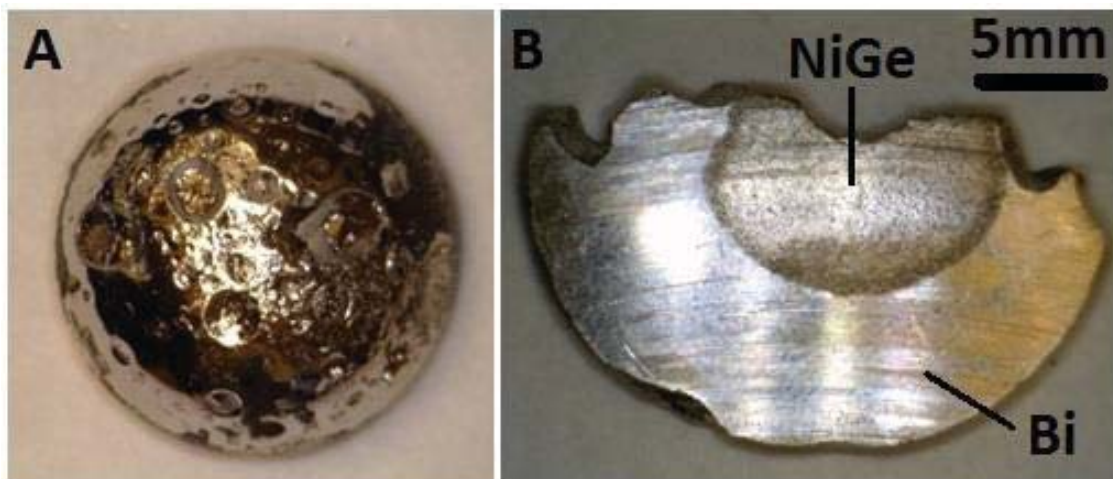
Eilertsen et al. *Acta Mater.* **60** (2012) 2178-2185.  
Chi et al. *Phys. Rev. B* **86**: 195209 (2012).

Chi et al. Phys. Rev. B **86**: 195209 (2012).

# Filled $\text{Co}_x\text{Ni}_{4-x}\text{Sb}_{12-y}\text{Sn}_y$ Skutterudites

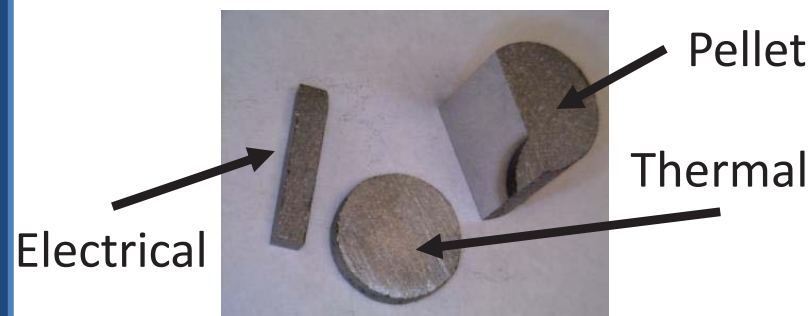
## Systems Investigated

- Ternary systems studied with combination of solidification and powder processing techniques.
- $\text{Ni}_4\text{Bi}_8\text{Ge}_4$ 
  - Shown below, skutterudite phase not obtained.
- $\text{Ni}_4\text{Sb}_8\text{Ge}_4$ 
  - Skutterudite phase not obtained.
- $\text{Ni}_4\text{Sb}_8\text{Sn}_4$



## Objectives

- Focus on finding a p-type skutterudite with improved ZT.
- Study behavior of the skutterudite  $\text{Co}_x\text{Ni}_{4-x}\text{Sb}_{12-y}\text{Sn}_y$ .
  - Grytsiv et. al has reported a  $\text{Ni}_4\text{Sb}_8\text{Sn}_4$  skutterudite system.
  - Parameters of study:
    - $x = \{0, 0.5, 1, 1.5, 2\}$
    - $y = \{3, 4, 5\}$
- Samples created from a melt/mill/hot press procedure.



S

- Ternary sy
- of solidific
- technique

- $\text{Ni}_4\text{Bi}_8\text{Ge}_4$
- Show
- obtai

- $\text{Ni}_4\text{Sb}_8\text{Ge}_4$
- Skutt
- $\text{Ni}_4\text{Sb}_8\text{Sn}_4$



$$\text{Co}_x\text{Ni}_{4-x}\text{Sb}_{12-y}\text{Sn}_y$$

Sample	Co	Sn	Lattice
#	Parameter		
	(x)	(y)	(Å)
1	0.0	4.0	9.113
2	0.0	5.0	9.128
3	0.5	5.0	9.126
4	1.0	5.0	9.118
5	1.5	5.0	9.123
6	2.0	5.0	9.104
7	2.0	4.0	9.109
8	2.0	3.0	9.087

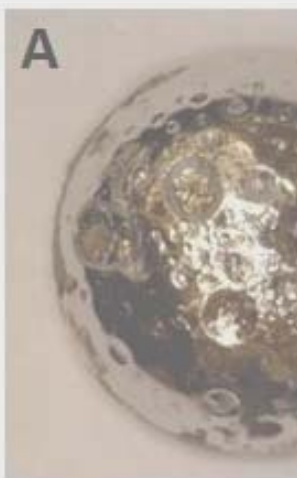
Objectives

$$\text{A}_z\text{Co}_2\text{Ni}_2\text{Sb}_8\text{Sn}_4$$

Sample	Filler	Level	Lattice
#	Parameter		
	A	(z)	(Å)
7	N/A	0.0	9.109
9	Ce	0.1	9.108
10	Dy	0.1	9.114
11	Yb	0.05	9.019
12	Yb	0.1	9.111
13	Yb	0.2	9.114

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- Ternary sy of solidific technique
- $\text{Ni}_4\text{Bi}_8\text{Ge}_4$ 
  - Show obtai
- $\text{Ni}_4\text{Sb}_8\text{Ge}_4$ 
  - Skutt
- $\text{Ni}_4\text{Sb}_8\text{Sn}_4$



Sample #	Co (x)	Sn (y)	Lattice Parameter (Å)
1	0.0	4.0	9.113
2	0.0	5.0	9.128
3	0.5	5.0	9.126
4	1.0	5.0	9.118
5	1.5	5.0	9.123
6	2.0	5.0	9.104
7	2.0	4.0	9.109
8	2.0	3.0	9.087

Objectives



Sample #	Filler A	Level (z)	Lattice Parameter (Å)
7	N/A	0.0	9.109
9	Ce	0.1	9.108
10	Dy	0.1	9.114
11	Yb	0.05	9.019
12	Yb	0.1	9.111
13	Yb	0.2	9.114



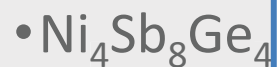
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- technique

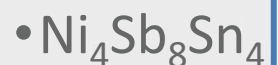


- Show

obtai



- Skutt



Sample	Co	Sn	Lattice
#	Parameter		
	(x)	(y)	(Å)
1	0.0	4.0	9.113
2	0.0	5.0	9.128
3	0.5	5.0	9.126
4	1.0	5.0	9.118
5	1.5	5.0	9.123
6	2.0	5.0	9.104
7	2.0	4.0	9.109
8	2.0	3.0	9.087

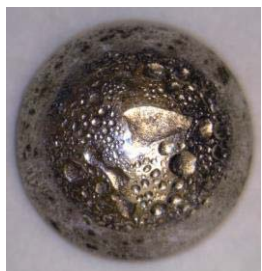
Objectives



Sample	Filler	Level	Lattice
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	A	(z)	(Å)
7	N/A	0.0	9.109
9	Ce	0.1	9.108
10	Dy	0.1	9.114
11	Yb	0.05	9.019
12	Yb	0.1	9.111
13	Yb	0.2	9.114

## ICP analysis of an ingot

- 2 Hr @ 1100°C (+20,-10°C /min)
- Silica crucible in He atmosphere
- <1% wt loss

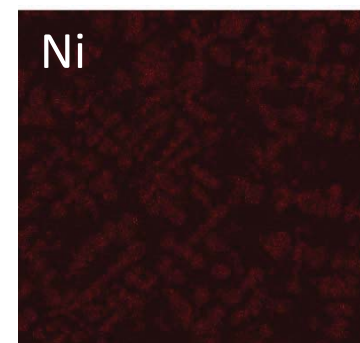
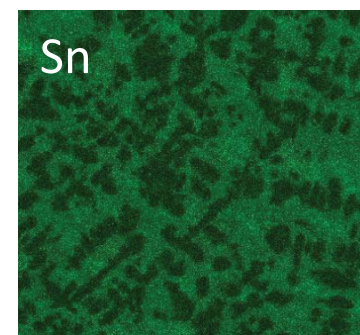
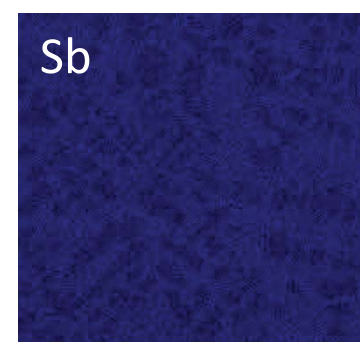
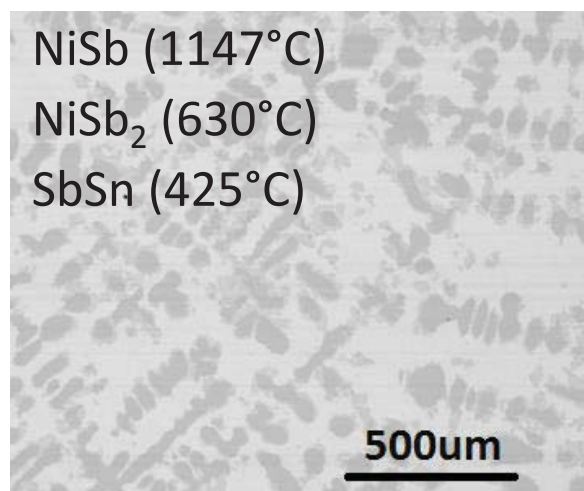


Target



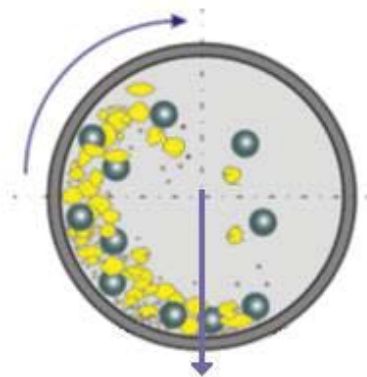
	at%	at%	at%	at%
Co	<b>9.4</b>	9.1	7.3	9.0
Ni	<b>15.6</b>	14.9	13.7	14.6
Sb	<b>43.7</b>	42.4	43.7	44.1
Sn	<b>31.2</b>	33.5	35.3	32.2
Ca	<b>0</b>	2e-4	7e-4	7e-4
Mg	<b>0</b>	1e-4	2e-4	2e-4
Na	<b>0</b>	3e-3	4e-3	4e-3

## EDS map of an ingot

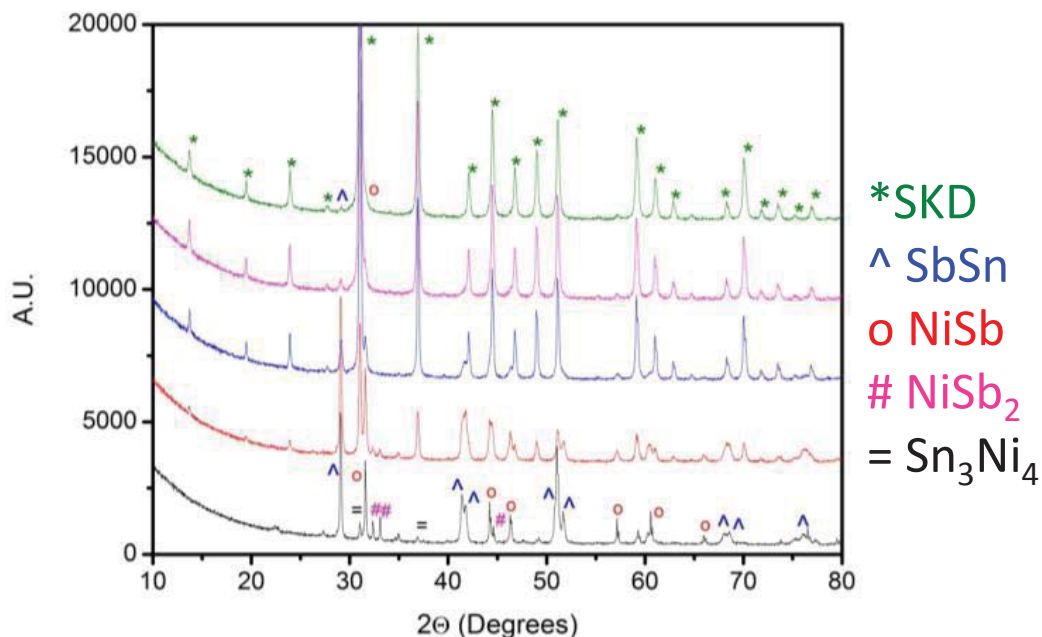


## Milling Details

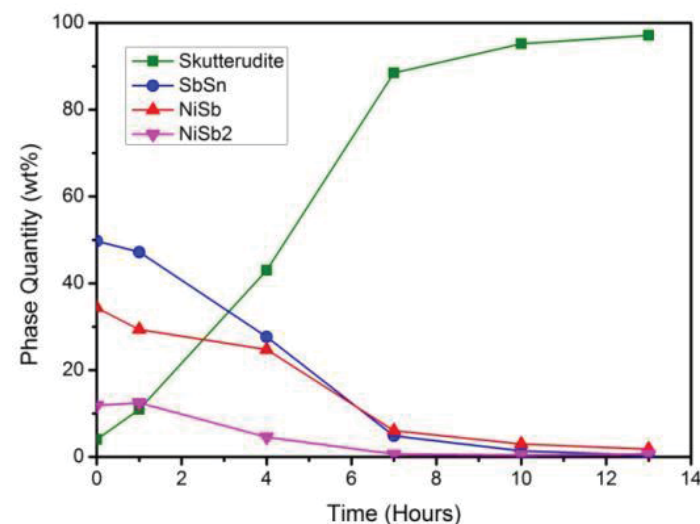
- Planetary mill
- 550 rpm
- Ball to powder weight ratio 3.8
- Ar atmosphere



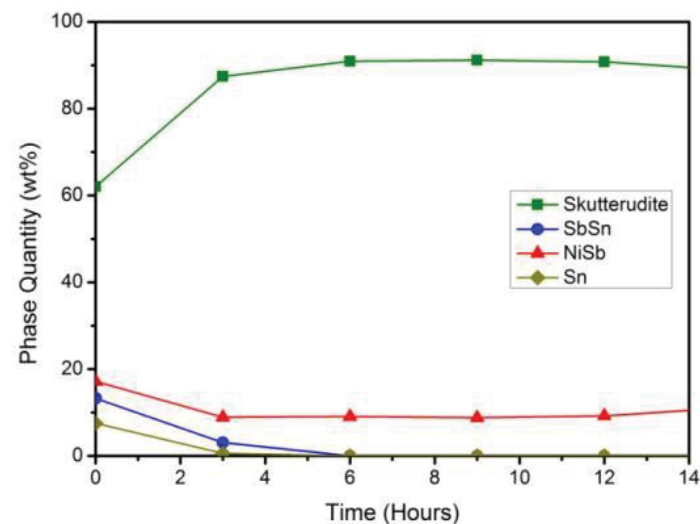
## Milling XRD



## Sample 1 Ni<sub>4</sub>Sb<sub>8</sub>Sn<sub>4</sub> Milling



## Sample 4 Co<sub>1</sub>Ni<sub>3</sub>Sb<sub>7</sub>Sn<sub>5</sub> Milling

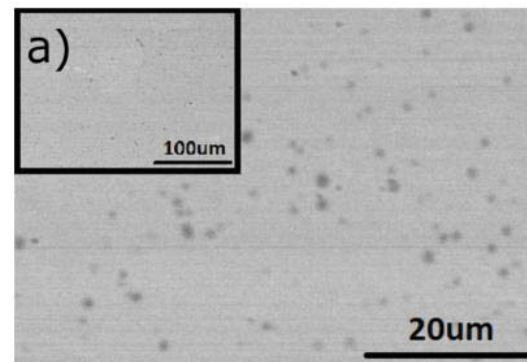




### Hot Pressed SEM

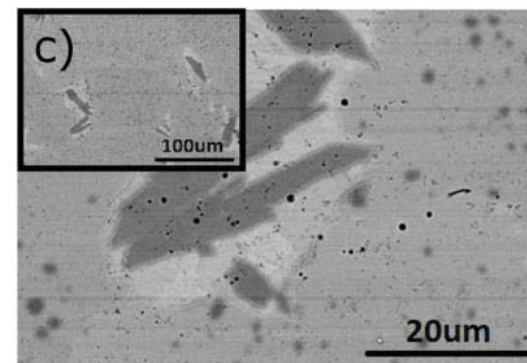
#### Sample 1 $\text{Ni}_4\text{Sb}_8\text{Sn}_4$

- NiSb (3.1wt%, 109nm cryst.) precip 1 $\mu\text{m}$ .
- SbSn (1.3wt%, 45 nm cryst.) precip 30  $\mu\text{m}$ .



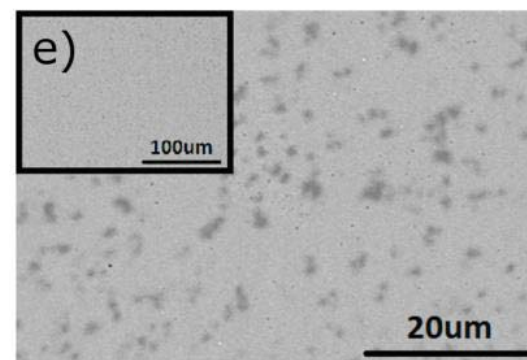
#### Sample 2 $\text{Ni}_4\text{Sb}_7\text{Sn}_5$

- NiSb (6.8wt%) precip 1 $\mu\text{m}$ .
- $\text{Ni}_3\text{Sn}_4$  (1.2wt%) precip 30  $\mu\text{m}$ .
- SbSn (1.4wt%) surrounding  $\text{Ni}_3\text{Sn}_4$ .



#### Sample 4 $\text{Co}_1\text{Ni}_3\text{Sb}_7\text{Sn}_5$

- NiSb (3.2wt%) precip 1 $\mu\text{m}$ .
- $\text{Ni}_3\text{Sn}_4$  (6.5wt%) precip 1 $\mu\text{m}$ .



## Rietveld Refinement

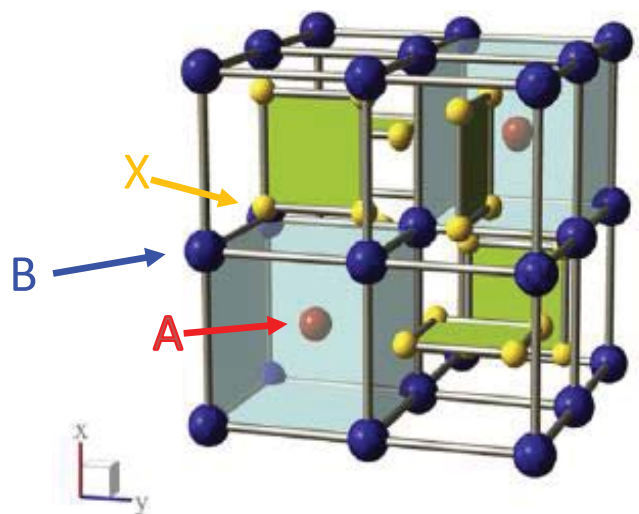
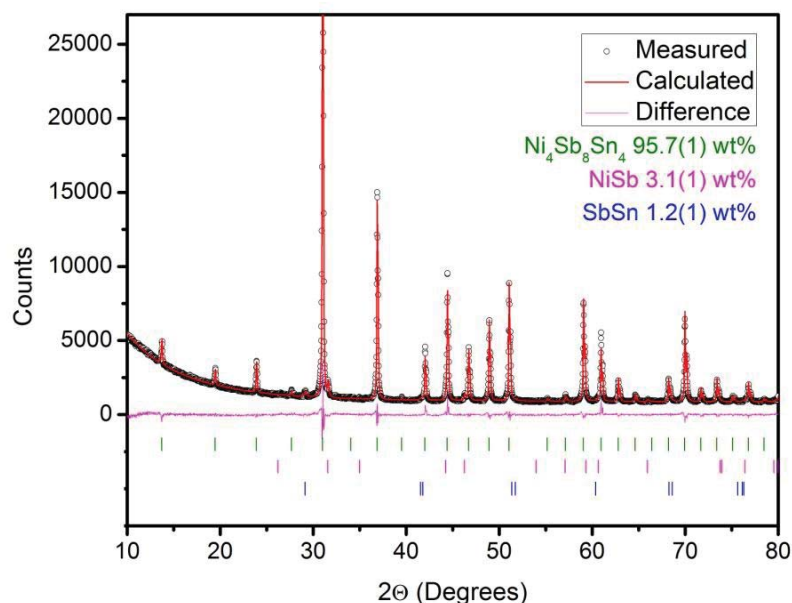


FIGURE: Eilertsen et al. Acta Mater. **60** (2012) 2178-2185.

## Hot Pressed Structure Refinement

Sample #	Skutterudite	Lattice (Å)	SKD (wt%)
	$A_\delta B_x B'_{4-x} X_{12-y} X'_y$		
1	Sn <sub>0.2</sub> Co <sub>0.0</sub> Ni <sub>4.0</sub> Sb <sub>8.5</sub> Sn <sub>4.4</sub>	9.113	96.65
2	Sn <sub>0.3</sub> Co <sub>0.0</sub> Ni <sub>4.0</sub> Sb <sub>7.9</sub> Sn <sub>5.1</sub>	9.128	87.38
3	Sn <sub>0.3</sub> Co <sub>0.6</sub> Ni <sub>3.4</sub> Sb <sub>7.2</sub> Sn <sub>4.7</sub>	9.126	94.97
4	Sn <sub>0.3</sub> Co <sub>1.2</sub> Ni <sub>2.8</sub> Sb <sub>8.3</sub> Sn <sub>5.4</sub>	9.118	89.25
5	Sn <sub>0.3</sub> Co <sub>1.5</sub> Ni <sub>2.5</sub> Sb <sub>7.0</sub> Sn <sub>4.7</sub>	9.123	91.33
6	Sn <sub>0.3</sub> Co <sub>2.4</sub> Ni <sub>1.6</sub> Sb <sub>9.4</sub> Sn <sub>5.8</sub>	9.104	80.08
7	Sn <sub>0.3</sub> Co <sub>2.1</sub> Ni <sub>1.9</sub> Sb <sub>9.1</sub> Sn <sub>3.7</sub>	9.109	93.64
8	Sn <sub>0.2</sub> Co <sub>2.1</sub> Ni <sub>1.9</sub> Sb <sub>9.0</sub> Sn <sub>2.6</sub>	9.087	98.20

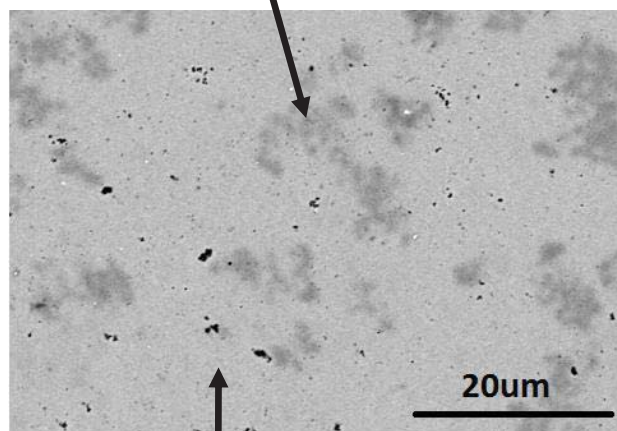
# Introduction

## Pressed $\text{Co}_2\text{Ni}_2\text{Sb}_7\text{Sn}_5$

Density 7.64 g/cm<sup>3</sup>  
99%

Phase	Wt%
$\text{Co}_2\text{Ni}_2\text{Sb}_7\text{Sn}_5$	82.6
$\text{Ni}_3\text{Sn}_4$	8.7
Sn	6.2

$\text{Ni}_3\text{Sn}_4$  (230°C)



$\text{Sn}_{0.5}\text{Co}_{2.4}\text{Ni}_{1.6}\text{Sb}_{9.7}\text{Sn}_{5.7}$

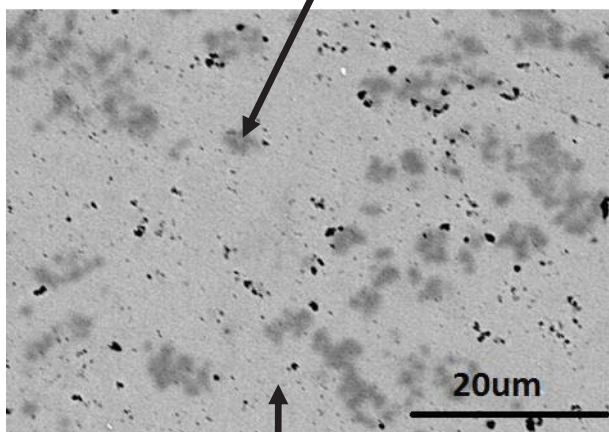
# Processing

## 200°C Anneal 72 Hrs

Density 7.25 g/cm<sup>3</sup>  
95%

Phase	Wt%
$\text{Co}_2\text{Ni}_2\text{Sb}_7\text{Sn}_5$	80.0
$\text{Ni}_3\text{Sn}_4$	11.9
Sn	7.6

$\text{Ni}_3\text{Sn}_4$  (230°C)



$\text{Sn}_{0.5}\text{Co}_{2.4}\text{Ni}_{1.6}\text{Sb}_{9.7}\text{Sn}_{5.7}$

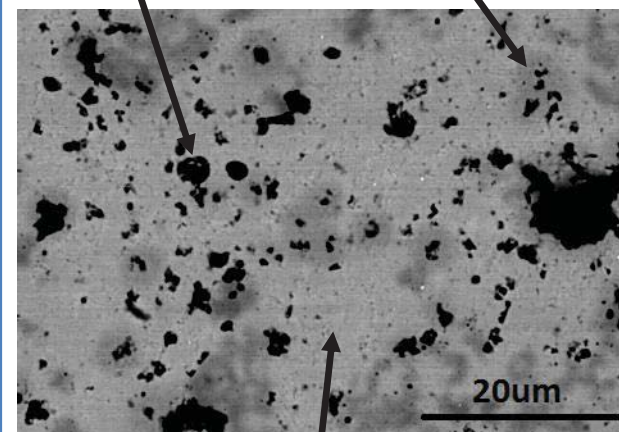
# Properties

## 400°C Anneal 72 Hrs

Density 6.75 g/cm<sup>3</sup>  
88%

Phase	Wt%
$\text{Co}_2\text{Ni}_2\text{Sb}_7\text{Sn}_5$	73.6
$\text{Ni}_3\text{Sn}_4$	14.7
Sn	10.0

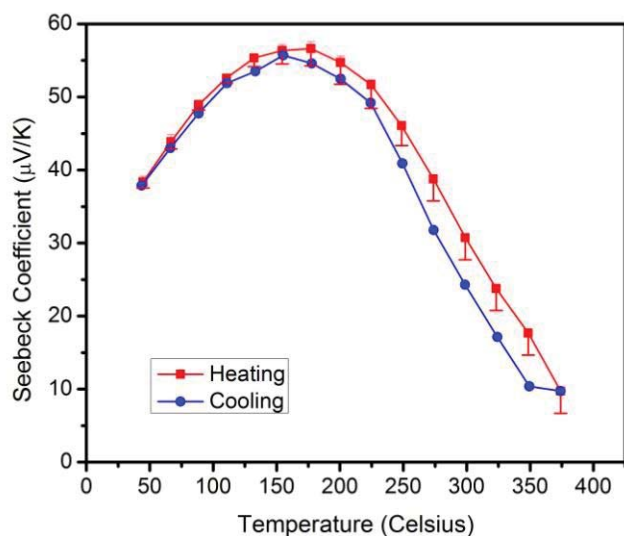
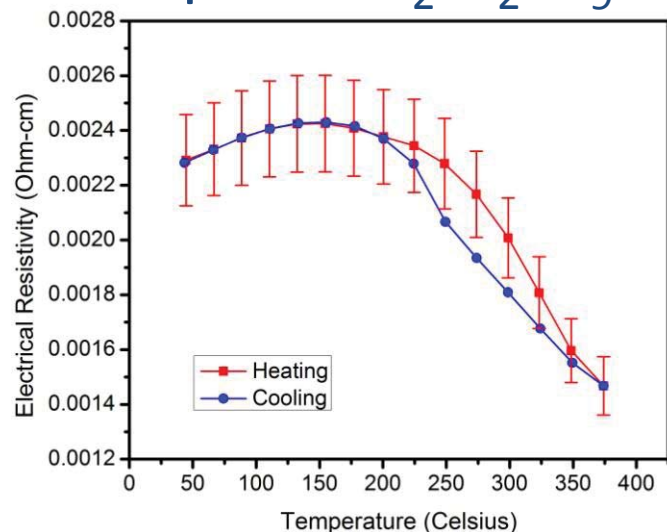
Porosity  $\text{Ni}_3\text{Sn}_4$  (230°C)



$\text{Sn}_{0.5}\text{Co}_{2.4}\text{Ni}_{1.6}\text{Sb}_{9.7}\text{Sn}_{5.7}$

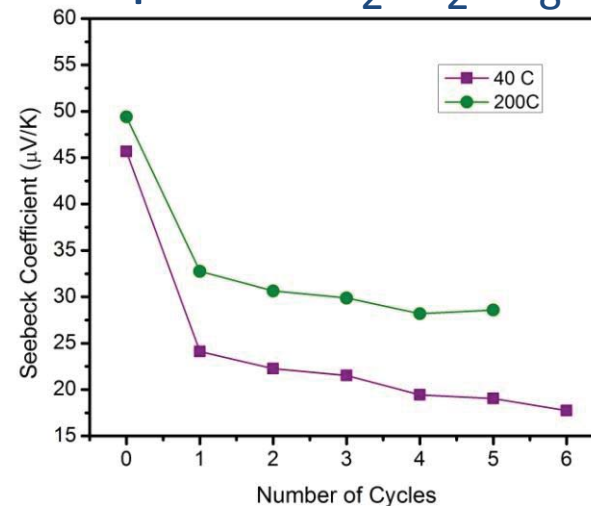
## Electrical Hysteresis

Sample 8  $\text{Co}_2\text{Ni}_2\text{Sb}_9\text{Sn}_3$

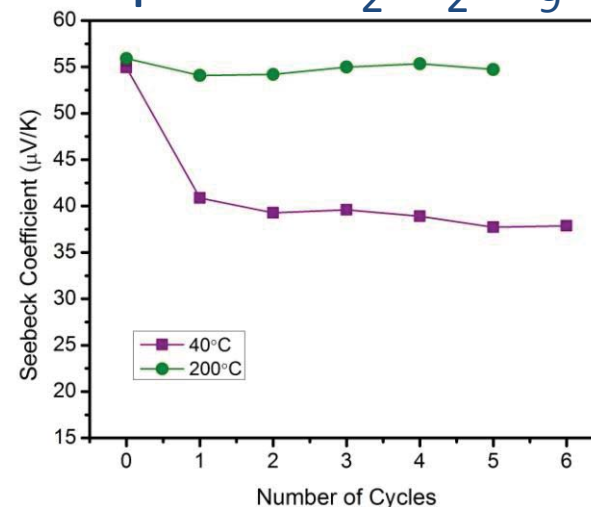


## Sample Stability

Sample 7  $\text{Co}_2\text{Ni}_2\text{Sb}_8\text{Sn}_4$



Sample 8  $\text{Co}_2\text{Ni}_2\text{Sb}_9\text{Sn}_3$



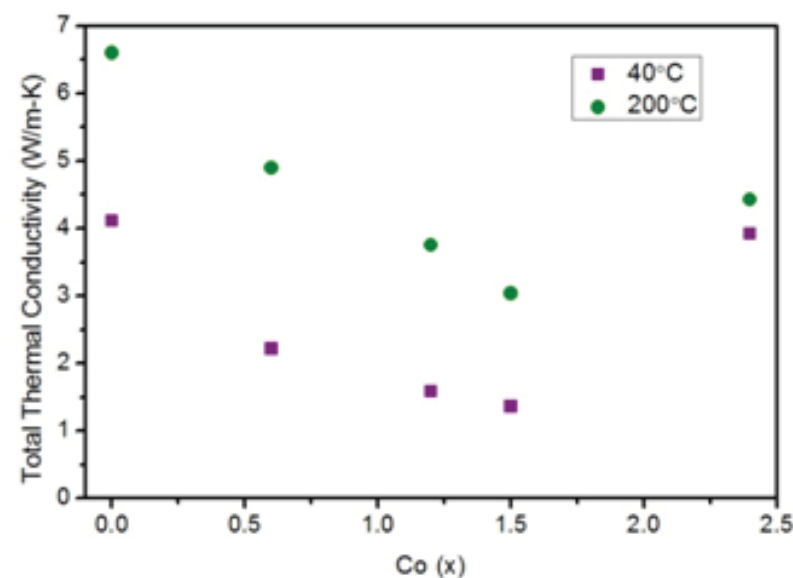
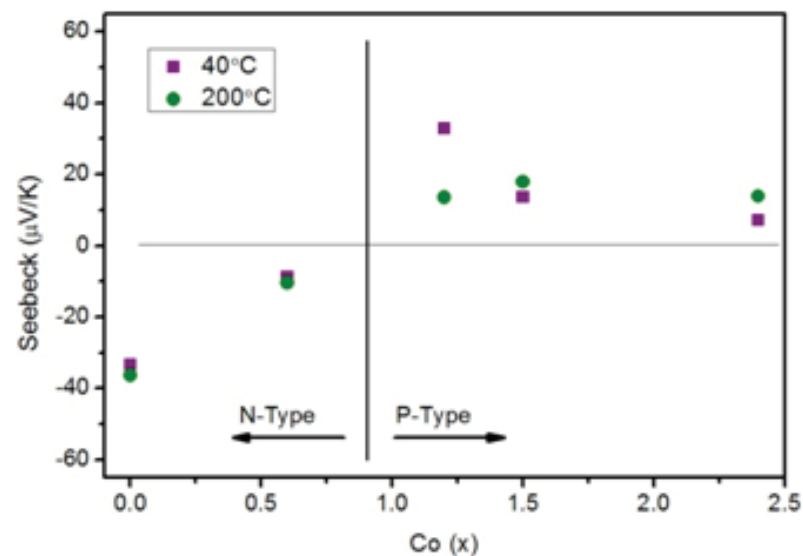


## Transport Properties- Unfilled (40°C)



Sample #	Co (x)	Sn (y)	Lattice Parameter (Å)	Seebeck Coefficient ( $\mu\text{V/K}$ )	Electrical Resistivity ( $\mu\text{Ohm} - \text{cm}$ )	Thermal Conductivity ( $\text{W/m-K}$ )
1	0.0	4.0	9.113	-40.7	233	4.7
2	0.0	5.0	9.128	-33.4	255	4.1
3	0.5	5.0	9.126	-8.7	560	2.2
4	1.0	5.0	9.118	32.9	784	1.6
5	1.5	5.0	9.123	13.7	449	1.4
6	2.0	5.0	9.104	7.1	233	3.9
7	2.0	4.0	9.109	17.7	540	2.5
8	2.0	3.0	9.087	37.9	2282	1.5

## Co (x) Study

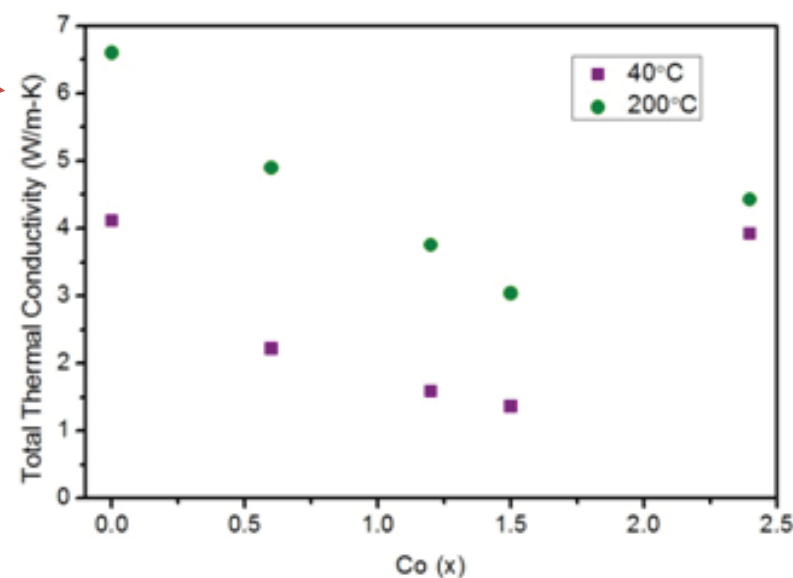
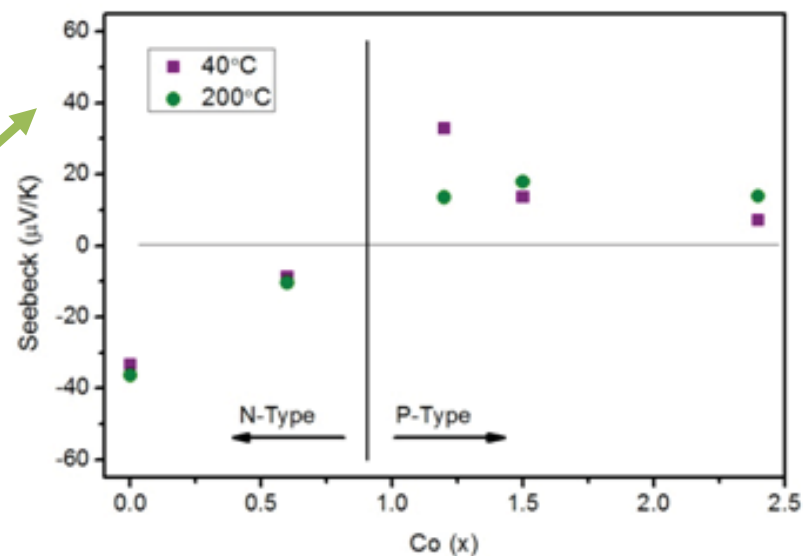


## Transport Properties- Unfilled (40°C)



Sample #	Co (x)	Sn (y)	Lattice Parameter (Å)	Seebeck Coefficient ( $\mu\text{V/K}$ )	Electrical Resistivity ( $\mu\text{Ohm} - \text{cm}$ )	Thermal Conductivity ( $\text{W/m-K}$ )
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8	2.0	3.0	9.087	37.9	2282	1.5

## Co (x) Study

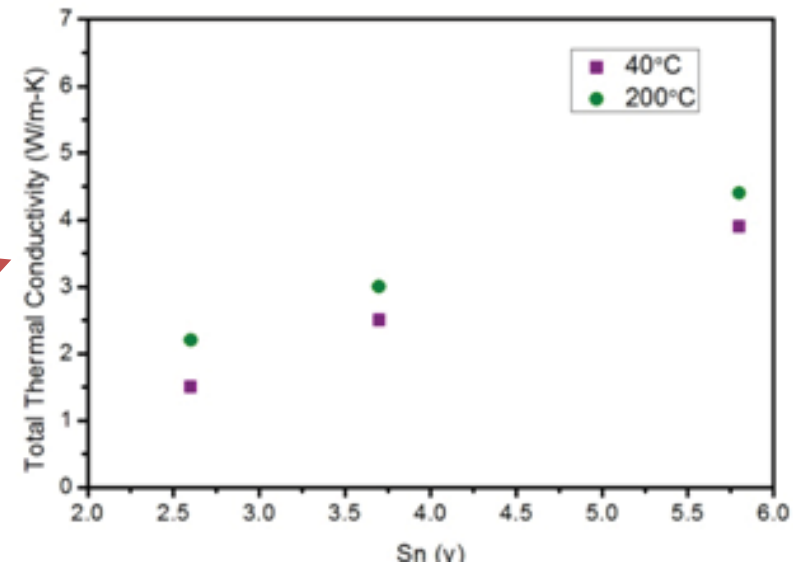
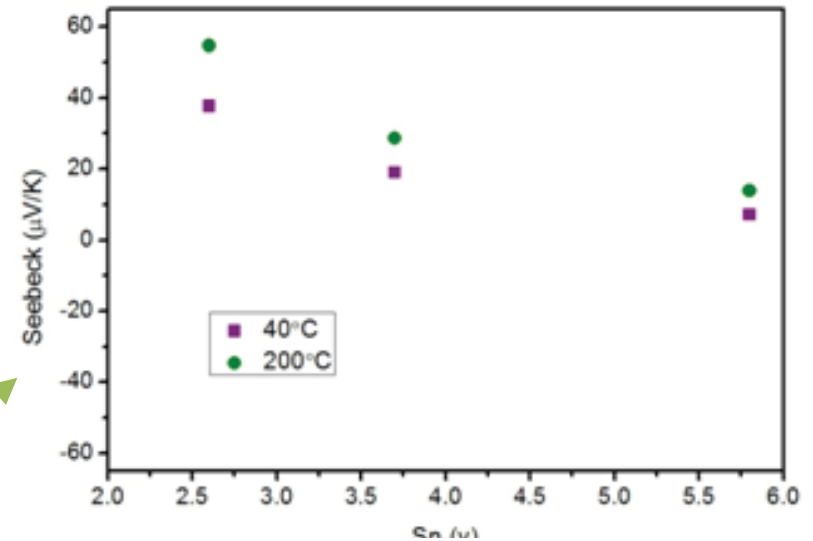


## Transport Properties- Unfilled (40°C)



Sample #	Co (x)	Sn (y)	Lattice Parameter (Å)	Seebeck Coefficient ( $\mu\text{V/K}$ )	Electrical Resistivity ( $\mu\text{Ohm} - \text{cm}$ )	Thermal Conductivity (W/m-K)
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7	2.0	4.0	9.109	17.7	540	2.5
8	2.0	3.0	9.087	37.9	2282	1.5

## Sn (y) Study

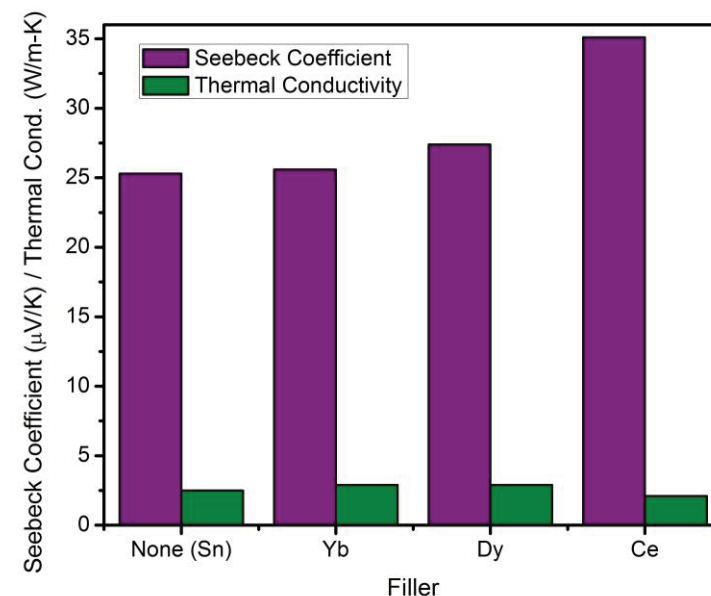
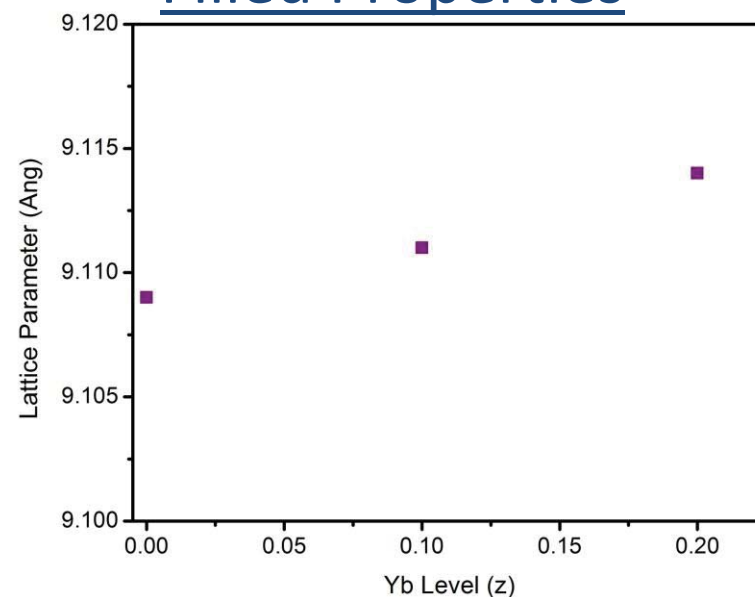


## Transport Properties- Filled (40°C)



Sample #	Filler	Level (z)	Lattice Parameter (Å)	Seebeck Coefficient ( $\mu\text{V}/\text{K}$ )	Electrical Resistivity ( $\mu\text{Ohm} - \text{cm}$ )	Thermal Conductivity (W/m-K)
7	N/A	0.0	9.109	25.3	659	2.5
9	Ce	0.1	9.108	35.1	1036	2.1
10	Dy	0.1	9.114	27.4	681	2.9
11	Yb	0.05	9.019	23.3	618	2.6
12	Yb	0.1	9.111	25.6	592	2.9
13	Yb	0.2	9.114	-	-	-

## Filled Properties



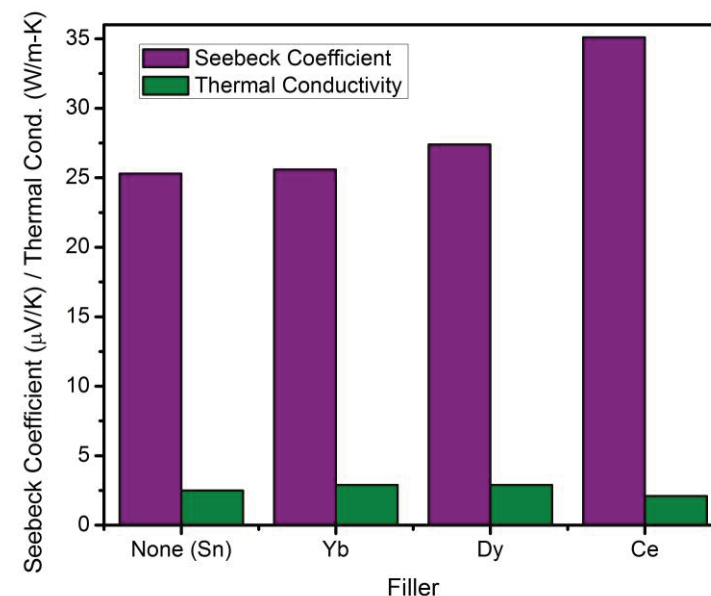
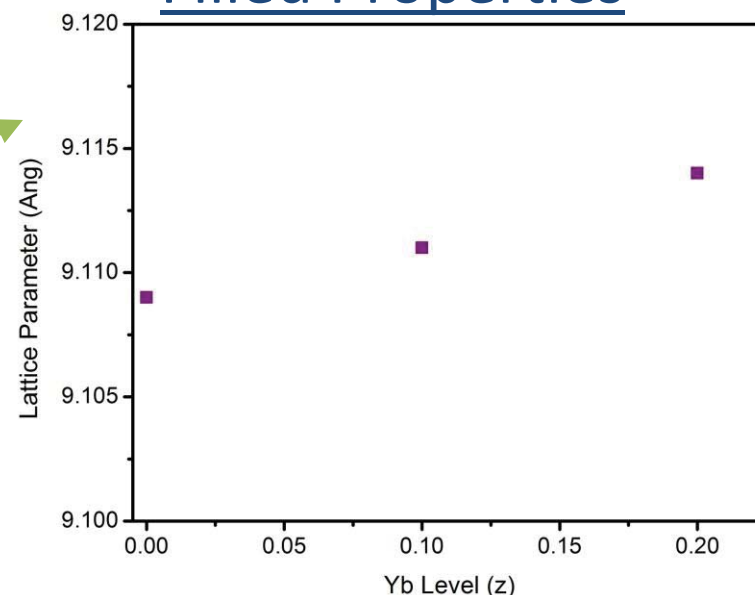


## Transport Properties- Filled (40°C)

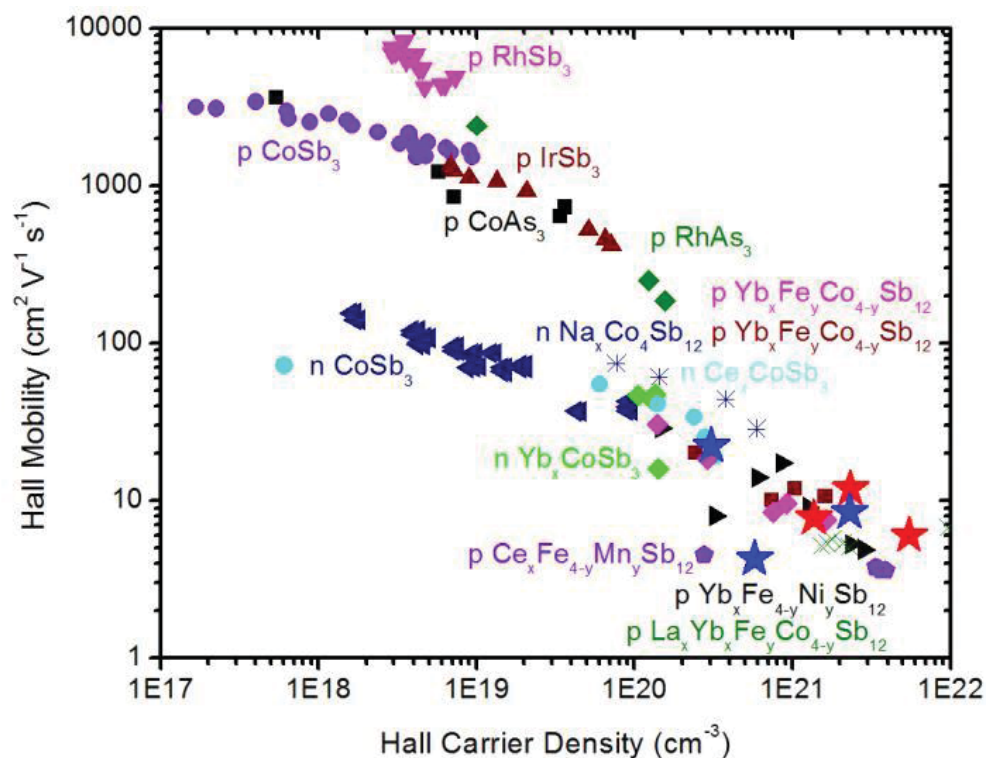


Sample #	Filler A	Level (z)	Lattice Parameter (Å)	Seebeck Coefficient ( $\mu\text{V}/\text{K}$ )	Electrical Resistivity ( $\mu\text{Ohm} - \text{cm}$ )	Thermal Conductivity (W/m-K)
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13	Yb	0.2	9.114	-	-	-

## Filled Properties



## Mobility and Carrier Comparison

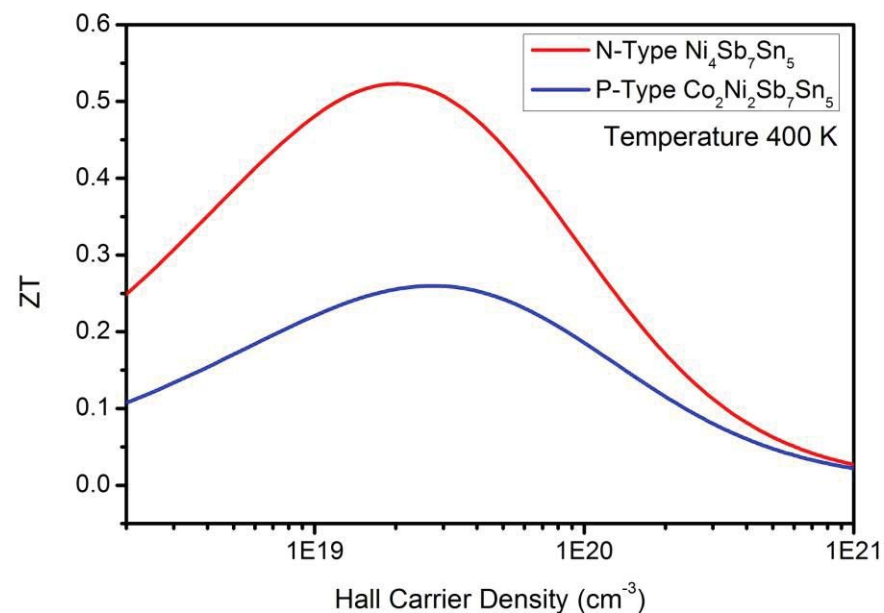


★ ★ This Work

Yb<sub>x</sub>CoSb<sub>3</sub>: L. Fu et al. Intermetallics (2013)  
 Ce<sub>x</sub>CoSb<sub>3</sub>: D. Morelli et al. Phys. Rev. B (1997)  
 Others: J.-P. Fleurial et al. Proc. XVI ICT (1997)

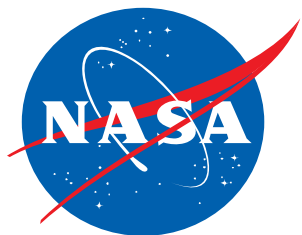
## S.P.B. Modeling

- Applied a single parabolic band model to the system
- Carrier mass ( $m/m_e$ )
  - N-Type: 5.48
  - P-Type: 1.48
- Optimal carrier density
  - N-Type:  $2.1\text{E}19 \text{ cm}^{-3}$
  - P-Type:  $2.7\text{E}19 \text{ cm}^{-3}$



## Conclusion

- The  $\text{Co}_x\text{Ni}_{4-x}\text{Sb}_{12-y}\text{Sn}_y$  skutterudite can be synthesized from a melt/mill/hot press schedule.
- Both n- and p-type conduction can be achieved by Co doping.
- System exhibits low thermal conductivity, but also low Seebeck coefficient.
- Thermoelectric performance of the system is hindered by large carrier densities and low carrier mobilities.
- Fillers improve Seebeck coefficient, but do not reduce thermal conductivity.



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